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(54) FLOWTHROUGH CUVETTE

(71) We, MICROMEDIC SYSTEMS, INC., a corporation organised and existing under the laws of the State of Delaware, United States of America, of Rohm and Haas Building, Independence Mall West, Philadelphia, Pennsylvania 19105, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The invention relates to cuvettes of the flowthrough type and to the production thereof.

There are various types of cuvettes commonly used for holding a liquid specimen or sample intended to undergo measurement, for example by means of a spectrophotometer. To this end, the known cuvettes generally consist of a transparent body made for instance of glass or quartz and comprising a cylindrical cavity for receiving the specimen. This cavity extends inside the cuvette over a given distance constituting the "optical path" travelled by the light beam passing through the sample during the measuring operation. The two ends of the cavity are delimited by transparent zones of the cuvette wall, which zones constitute inlet and outlet windows for the said beam.

This cavity communicates with the outside of the cuvette via two transverse channels which are respectively connected to the two ends of the cavity and enable the sample to be inserted in the cavity and to be removed therefrom after the measurement has been effected. The cylindrical cavity thus forms a central elongated limb of a doubly-bent continuous passage.

One type of commonly used cuvette consists of a quartz or glass tube, which is bent at two places to form the said passage. The central cavity of this passage is thus delimited at its ends by curved portions of the wall of the twice-elbowed tube forming the cuvette. Consequently, the said inlet and outlet win-

dows for the measuring beam are also curved, thus causing light deviation, and hence a risk of affecting the accuracy of the measurement. Further, this mode of production, which consists in bending a tube, does not make it possible to determine in a very accurate and reproducible manner the length of the said optical path for the beam of light passing through the liquid specimen. Now, even a relatively small variation of this length, between different cuvettes, is liable to cause measurement errors.

In a known variant of the above-described known cuvette, the said windows are flattened to obviate the cited drawbacks. To this end, the doubly-bent tube is softened by heating and two flat plates are pressed against the curved windows to flatten them and to adjust the distance between them. Such indirect deformation of the internal surface of the windows, by acting on their outside faces, does not however make it possible fully to overcome the above-mentioned drawbacks of the type of cuvette.

In a second known variant of this type of known cuvette, the twice-elbowed tube is cut out so as to remove the said curved portions defining the two ends of the cavity and two flat transparent plates are fixed in their stead so as to form flat windows. This cutting operation obviously requires a very high degree of accuracy so as to set in a precise manner the distance between the said plates and hence the optical path of the beam. The cuvette according to this second variant does nonetheless enable the above-mentioned drawbacks to be overcome to a large extent, provided this accuracy is ensured. However, it has another drawback due to the fact that the said plates are disposed transversely to the central cavity and thus form corners, approximately at right angles, at the two ends of this cavity, such corners forming stagnant zones in which a residue of the liquid tends to remain after discharge of the sample. Such a residue from one sample to the next can however significantly affect the accuracy of

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the measurement.

Besides the above-mentioned drawbacks, the production of this type of known cuvette, as well as of the two described variants, gives rise to problems which become more and more difficult to solve as the size of the cuvette decreases. Thus, it has not been possible so far to produce cuvettes as described having the very small dimensions needed to perform measurements on liquid micro-specimens.

Another known cuvette, which is intended for micro-samples, comprises a block traversed by a capillary bore forming the central measuring cavity and transparent plaquettes mounted at the ends of this cavity to form the previously mentioned windows for the entry and exit of the light beam serving to effect the measurement. The lateral bores are moreover provided in the block so as to connect the two ends of said central cavity with the exterior and to thus form a doubly-bent passage with the central bore. This type of capillary cuvette allows the length of the optical path of the light beam traversing the cuvette to be fixed quite accurately. However, the doubly-bent passage, delimited by the walls of said bores, likewise has right-angled corners and sudden changes of direction at both ends of the bore forming the central cavity. This results in the previously mentioned drawback of stagnant zones leading to sample residues which can render the measurements inaccurate from one sample to the next.

A different type of known cuvette is the so-called "thin-layer cuvette", wherein the liquid is admitted between two transparent plates mounted at a slight distance from each other and forming the said windows for entry and exit of the light beam. The use of this type of cuvette is however limited to effecting measurements on opaque liquids, such as non-diluted blood, due to the fact that the optic path through the thin liquid layer is very short.

We have now found a cuvette of relatively simple construction and ease of manufacture in which many of these drawbacks can be overcome.

According to the invention there is provided a flow-through cuvette which comprises a piece or pieces of transparent material sealingly mounted and sandwiched between plates said piece(s) having an edge or edges so shaped that opposing edges of the transparent piece(s) together with both inner opposing faces of the plates define a flow-through passage comprising (a) a central length, the longitudinal axis of which does not pass through the plates, and (b) fluid inlet and outlet passages whose axes diverge from said longitudinal axis and connect the ends of the central length to the cuvette surface.

A continuous passage, as described above,

may be referred to in this specification as being doubly bent.

Preferably the edge(s) of said piece(s) are so shaped that the transition from each inlet and outlet (i.e. branch) passage to the central length is gradual so that streamline fluid flow through the cuvette is possible.

The piece or pieces of transparent material may comprise a pair of plates lying in a common plane with their shaped edges facing each other. Preferably they are spaced a predetermined distance apart.

In another embodiment of the invention the branch channels extend in opposite directions from the ends of the central length so that they open on opposite surfaces of the cuvette. Such a configuration provides a continuous passage through the cuvette which has a general s-shape.

In a preferred embodiment at least one of said edges of the transparent piece or pieces is so shaped as to provide at least one length of enlarged diameter in the central length.

To facilitate the provision of accurate, known dimensions in the central length, the invention also provides a cuvette in which the shaped edge(s) are formed by cutting a doubly-bent slot in a single plate, dispensing with the need for accurately arranging a pair of pieces.

The invention also provides multiple cuvettes, i.e. cuvettes having more than one doubly-bent passage therethrough. Such cuvettes may be formed by arranging more than two plates parallel to each other and sandwiching a shaped transparent piece(s) between either at least two pairs of, or between each, opposing face(s) of the parallel plates.

Particularly useful among multiple cuvettes is the double cuvette in which one passage receives a sample which may be compared, in measurement, with a reference sample in the other passage.

The cuvettes of the invention may be used in various measuring techniques such as spectrophotometry. They may also be used to determine the absorption or fluorescent properties of specimens. In the latter case, the lateral layers of the cuvette should be transparent in addition to the centre piece(s) in order to allow measurement of the fluorescent properties of the sample, perpendicularly to the longitudinal axis of the central length.

The invention also provides a method of manufacturing flowthrough cuvettes having a double-bent continuous passage comprising a central length and inlet and outlet branch passages extending therefrom to the surface of the cuvette.

The method comprises so shaping a piece or pieces of transparent material and sealingly mounting and sandwiching the piece(s) between plates that opposing shaped edges

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of the transparent piece(s) define the doubly-bent passage.

At least one transparent plate is shaped so as to provide at least one pair of edges corresponding to the desired double-bent path of the passage and a gradual transition between the central length of the passage and the transverse branch passages. The shaped edges are then arranged in a common plane and spaced a predetermined distance apart with their shaped edges facing each other so as to provide a central length of predetermined dimensions.

In a preferred embodiment the shaped edges are formed by cutting out a double-bent slot corresponding to the desired longitudinal profile of the passage in a single plate and rounding off the edges of the slot so as to provide a gradual transition between the central length and the transverse branch passages. The plate so formed is then sealingly mounted and sandwiched between plates and the resultant composite block is cut to expose the openings of the branch passages.

Some preferred embodiments of the cuvette of the invention will now be more particularly described by reference to the accompanying drawings, in which,

Figure 1 shows in perspective, partly broken away, an optical cuvette according to a first form of embodiment.

Figure 2 is a front view of the cuvette according to Figure 1, with an outer plate removed.

Figures 3a to 3d show, in section, four variants of the cuvette according to Figure 1.

Figure 4 represents, in longitudinal section, a double cuvette according to a second form of embodiment.

Figures 5a to 5b schematically illustrate two steps of the manufacturing method.

Figures 6a and 6b schematically illustrate two steps of a variant of the manufacturing method.

Figures 7 and 8 respectively show a longitudinal and a transverse sectional view of a third form of embodiment.

The cuvette represented in Figures 1 and 2 consists of a stratified block 1, made up of three layers. Two small rectangular plates 2a and 2b form the outer layers of this block 1 and a pair of similar plates 3a and 3b having the general form of the letter L are arranged in spaced head to foot relationship in the same plane so as together to form its intermediate layer.

As will be seen in Figure 1, an elongated central length or cavity 4 extends inside the block 1 along the mean longitudinal axis 0-0 representing the optical path and the ends of this cavity respectively communicate with two branch channels 5 and 6 which extend on opposite sides of this axis 0-0 and at right angles to the latter, as far as the opposite

faces A and B of the block 1 (see Figure 2). As may moreover be seen in Figure 1, this cavity 4 and these channels 5 and 6 thus constitute a continuous doubly-bent passage which extends through the block 1, in the intermediate layer of the latter.

This continuous passage 5-4-6 has a rectangular cross-section with a first pair of opposite sides respectively defined by the internal edges of the intermediate pair of plates 3a and 3b and a second pair of opposite sides respectively defined by the outer plates 2a and 2b. Thus, as will be observed from Figure 1, the inner edges of the plates 3a and 3b comprise three consecutive surfaces 7a, 8a, 9a and 7b, 8b, 9b, respectively, which form pairs of opposite surfaces, i.e. 7a and 7b, 8a and 8b, 9a and 9b, which pairs define two opposite sides of the channel 5, of the cavity 4 and of the channel 6, respectively.

These surfaces 7a, 8a, 9a and 7b, 8b, 9b are machined and polished, before the plates 3a and 3b are mounted in the block 1, so as to properly round the angles respectively formed between the successive surfaces 7a and 8a, 8a and 9a, 7b and 8b, and 8b and 9b. This rounding of the said angles enables any abrupt deviation along the said doubly-bent passage 5-4-6 to be eliminated by producing a gradual transition between the cavity 4, on the one hand, and the channels 5 and 6, on the other hand, thus eliminating any stagnant zone during the flow of liquid through this doubly-bent passage.

It thus becomes possible completely to discharge from the described cuvette each liquid sample that has served for the desired optical measurement. This discharge can be done by inserting into the cuvette another liquid thereby to force all of the liquid of said sample out of the cavity 4, without leaving any residue thereof in this cavity. In most cases, this other liquid may be the liquid sample which is to be subjected to the next measurement.

The structure of the described cuvette, in the form of a stratified block made up of four plates 2a, 2b, 3a and 3b, thus enables the cavity 4, as well as the channels 5 and 6, to be defined by the surfaces 7a, 8a, 9a and 7b, 8b, 9b which are readily accessible and which can thus be machined and polished without difficulty before assembling the cuvette. This not only enables the said passage to be given an outline which is favourable for complete discharge of each sample, but also to delimit the cavity 4 at its ends by windows having a flat polished surface and arranged at a precise predetermined distance from each other.

The plates 3a and 3b forming the intermediate layer are made of a transparent material, e.g. glass, quartz, or a plastics material such as acrylic resin. This material is selected in dependence on the wavelength of the light

intended to perform the required optical measurement, such that this material be transparent to the light having this wavelength. The side plates 2a and 2b are made of the same material as the intermediate plates 3a and 3b. Since the side plates 2a and 2b are not intended to be passed through by the light used to perform the measurement, in particular in a spectrophotometer, these plates are preferably blackened to reduce lateral light diffusion.

The assembly of the cuvette can be achieved in any suitable manner. In this instance, the plates 3a and 3b may be assembled with the side plates 2a and 2b by heat bending causing localized fusion of the adjacent surfaces of these plates.

The described cuvette is particularly suitable for optical measurements in a spectrophotometer and the mean axis 0-0 will then coincide with the optical axis of the spectrophotometer light beam.

The described form of embodiment can obviously be modified in various ways, with a view to adapting it better to the particular use to which the cuvette is to be put in each case.

Thus, for example, the channels 5 and 6 may be ground conically, after assembly of the described cuvette, to facilitate connection of these channels to a feed tube and to a discharge tube for the samples. This conical grinding, illustrated in dash-dotted lines in Figure 2, may form an angle of 5° in relation to the respective axes of the channels 5 and 6.

Further, it is not necessary for the stratified block constituting the cuvette to be rectangular, as described. Thus, the side plates 2a, 2b and the intermediate plates 3a and 3b may have different outlines like those shown by way of example in Figures 3a to 3d. These latter figures illustrate moreover some variants for the said doubly-bent passage consisting of the cavity 4 and of the channels 5 and 6.

Thus, for example, in the variants illustrated in Figures 3c and 3d, the cavity 4 further comprises at least one zone, 10 and 11, 12 respectively, which is located at a greater distance from the mean axis 0-0. Microbubbles of gas, which are often present in liquid specimens, can thus rise in the cavity 4 and accumulate in such a zone, which is located outside the axial measurement zone as such. It thus becomes possible to obviate the errors due to the presence of these microbubbles in the specimen during the optical measuring operation.

Figure 4 shows a cuvette of similar structure to that shown in Figures 1 and 2. However, the laminar block 1 is here made up of five layers and forms a double cuvette comprising two similar cavities 4 and 4', with their associated channels 5, 6 and 5', 6'. The block forming this double cuvette comprises plates 2a, 2b and 3a, 3b which define the

cavity 4 and the channels 5 and 6, as already described in relation to Figures 1 to 3. To form the second cavity 4' and associated passages 5' and 6', this block moreover comprises a further pair of intermediate plates 3a' and 3b', similar to the plates 3a and 3b described above, as well as a further side plate 2c similar to the plates 2a and 2b. The central plate 2b thus serves to define at the same time a part of the passage 5-4-6 and of the similar passage 5'-4'-6'.

The cavities 4 and 4' of the described double cuvette are thus arranged in identical manner and can for example be used to respectively receive a liquid specimen intended to be subjected to a measurement and a standard specimen used for a comparative measurement. The described double cuvette can thus be used to advantage to carry out measurements in a spectrophotometer of the well-known double beam type.

Figures 5a and 5b illustrate schematically two operations of a method of manufacturing a cuvette such as the one in Figure 1. As is apparent from Figure 5a, a glass or quartz plate 3 is first cut by means of an ultrasonic cutting tool 13 which has an S-shaped outline corresponding to that of the desired passage 5-4-6 so as to obtain a pair of identically shaped half-plates each comprising an edge formed by the three surfaces 7a, 8a, 9a and 7b, 8b, 9b, respectively. As will be observed from Figure 5b, several of these shaped plates are then placed side by side, and subjected to a polishing operation by means of a polishing tool 14.

The inner surfaces of the said windows through which the light beam is intended to pass are thus rendered perfectly flat and smooth and these surfaces may moreover be very accurately situated in all of the plates thus shaped.

The cuvette is finally assembled as shown in Figure 1 by juxtaposing in spaced head to foot relationship in the same plane, a pair of shaped and polished plates thus produced, e.g. in a jig having the initial dimensions of the plate 3, by disposing on opposite sides of this pair side plates 2a and 2b (see Figure 1) and by finally heat bonding the contacting surfaces between each of the plates 2a and 2b, on the one hand, and the plates 3a and 3b, on the other hand.

Figures 6a and 6b illustrate a variant of the described method. As is apparent from Figure 6a, use is also made of an ultrasonic cutting tool 17 having an outline in the form of an S corresponding to the desired profile of the passage 5-4-6. However, in this instance, the plate 3' is wider than the tool 17 so that this plate 3' remains in one piece after the cutting operation and comprises a central zone cut in the form of an S. As shown in Figure 6b, a polishing tool 18 is then used to

polish those parts of the surfaces 7b and 9a, which define the ends of the cavity 4, where the exit windows for entry and exit of the beam are to be located.

5 The plate 3', thus shaped and polished, is then assembled by fusion bonding, as already described, with two side plates of the same size to form a stratified block which is wider than the final cuvette-forming block. The longitudinal edges of the three plates forming this block are then cut, as indicated by dash-dotted lines in Figure 6b, so as to clear the inlet and outlet openings of the channels 5 and 6 and to give to the stratified block the desired final dimensions of the cuvette. There is thus produced a cuvette similar to that illustrated in Figure 1, but having a cavity 4 with a profile similar to that shown in Figure 3c. This variant according to Figures 6a and 6b facilitates the assembly of the stratified cuvette-forming block by obviating the need for accurately spaced juxtaposition in head to foot relationship of the plates 3a and 3b forming the intermediate layer, so as to obtain the exact desired length of the cavity 4.

It should be noted that the methods described above by way of example lend themselves to the manufacture on a large scale of cuvettes according to the various forms of embodiment of the invention, whilst ensuring very high accuracy and obviating the previously mentioned drawbacks of the known cuvettes. Indeed, these methods have enabled cuvettes according to Figure 1 and to Figure 4 to be manufactured very accurately and with very small dimensions, such as are needed to perform measurements on liquid micro-specimens, the overall dimensions of these cuvettes being at most of the order of 1 to 2 cm.

The outline of the doubly-bent passage may obviously have various forms other than those described and may be obtained by other means than an ultrasonic cutting tool, as described above. This outline may thus be obtained by any suitable machining means or by moulding.

Figures 7 and 8 represent a further embodiment of a double cuvette comprising two cavities 4 and 4' having a profile similar to that of the variant according to Figure 3c. This profile is defined by two pairs of plates 3a, 3b and 3a', 3b', which are respectively mounted between plates 2a, 2b and 2c (see Figure 8).

However, as may be seen from Figures 7 and 8, the said pairs of plates are turned around by 180° with respect to each other, around a vertical axis in the present case. An arrangement is thus obtained, wherein the transverse channels 5 and 5', as well as 6 and 6', respectively extend to diagonally opposite locations on the same sides of the cuvette. In view of the relatively small width of the

cuvette, this arrangement provides the advantage of considerably increasing the spacing between the channels which open on to the same face of the cuvette and of thus facilitating connection of the channels 5, 5' and 6, 6' to outside conduits, as described below.

In this embodiment, the cuvette is further provided with a pair of connecting blocks 19a and 19b made of glass, of plastics material or of metal, for example. These identical blocks are respectively mounted on the opposite faces of the cuvette, on to which the transverse channels 5, 5' and 6, 6' open, this assembly being achieved by gluing, welding or mechanical fastening for example.

Each of the blocks 19a and 19b comprises two connecting channels, 20, 20' and 21, 21', respectively, each aligned with one of the transverse channels 5, 5' and 6, 6' and each having a cross-section which varies progressively from a rectangular section, corresponding to that of the opposite transverse channel, up to a circular section communicating with a conical cavity 22, 22' and 23, 23', respectively, leading to the external surface of the corresponding block 19a or 19b.

These blocks 19a and 19b serve for the connection of conduits 24, 24' and 25, 25', respectively, and allow admission and discharge of the liquid samples which are to undergo optical measurement, in the central cavities 4 and 4' of the cuvette.

In the present instance, each of the conduits 24, 24' and 25, 25' has an enlarged conical end portion sealingly mounted in the corresponding cavity 22, 22' and 23, 23', so that its internal section is situated so as to form a prolongation of the corresponding connecting channel 20, 20' and 21, 21', respectively. These conduits are moreover mounted by means of shaped clamping plates 26a and 26b which are respectively fixed on to the opposite external faces of the blocks 19a and 19b. As may be seen from Figures 7 and 8, tightening springs 27, 27' and 28, 28', which are respectively mounted in recesses provided for this purpose in the plates 26a and 26b, each press against the enlarged end of one of the conduits 24, 24' and 25, 25', via a washer 29, 29' and 30, 30', respectively. These four conduits are thus connected in a simple manner to the described double cuvette, via the connecting blocks 19a and 19b having the channels 20, 20' and 21, 21' which provide a gradual transition of the flow cross-section at the outer end of the transverse channels 5, 5' and 6, 6' respectively associated with the central cavities 4, 4' of the passages of the double cuvette.

The double cuvette described above and shown in Figures 7 and 8 is further equipped with a heating device adapted to maintain the cuvette, and hence the liquid contained therein, at a desired temperature. This

device comprises an electrical resistance 31 coiled around the cuvette, together with its connecting blocks 19a and 19b, in a zone extending practically over the whole length of the central cavities 4 and 4' of the cuvette. This resistance is supplied with current from an electrical source 32 (see Figure 8) which is controlled in a well known manner by means of a thermostatic regulator (not shown) associated with a temperature measuring member 33 mounted in the middle of the median plate 2b situated between the two passages 5-4-6 and 5'-4'-6' of the described double cuvette.

Maintenance of the temperature of the samples at a constant value, by means of a heating device such as described above by way of example, is advantageous for various optical measurements, such as those intended for determining the activity of enzymes; in that case, the temperature may be maintained at 37°C for example. Heating of the cuvette may moreover obviously be ensured by any other conventional means, such as a thermostatically controlled circuit of heating liquid, arranged in any suitable manner allowing the samples contained in the cuvette to be maintained at the desired temperature.

It is understood that all the other cuvettes which have been described may likewise be equipped with heating means such as are mentioned and are described above.

When the cuvette is not provided with heating means, it may nevertheless be useful to equip it with means for measuring the temperature of the samples in order to be able to take it into account when effecting the optical measurements.

It may be noted that a cuvette according to the invention may be readily manufactured very accurately and in large series, without its dimensions playing a critical role; the manufacture of so-called "capillary" cuvettes in accordance with the invention thus does not present any particular problem.

WHAT WE CLAIM IS:—

1. A flowthrough cuvette which comprises a piece or pieces of transparent material sealingly mounted and sandwiched between plates said piece(s) having an edge or edges so shaped that opposing edges of the transparent piece(s) together with both inner opposing faces of the plates define a flow-through passage comprising (a) a central length, the longitudinal axis of which does not pass through the plates, and (b) fluid inlet and outlet passages whose axes diverge from said longitudinal axis and connect the ends of the central length to the cuvette surface.

2. A cuvette as claimed in claim 1 wherein the edge(s) of said piece(s) are so shaped that the transition from each inlet and outlet passage to the central length is gradual so that streamline fluid flow through the

cuvette is possible.

3. A cuvette as claimed in claim 1 or 2, wherein the inlet and outlet passages extend in opposite directions from the ends of the central length so that the passage is generally s-shaped between openings on opposite outer faces of the cuvette.

4. A cuvette as claimed in any preceding claim wherein at least one of the shaped edges is such as to provide at least one length of enlarged diameter in the central length.

5. A cuvette as claimed in any preceding claim wherein the central pieces of transparent material comprise a pair of plates lying in a common plane and spaced a predetermined distance apart with their shaped edges facing each other.

6. A cuvette as claimed in claim 5 wherein said edges of the pair of plates are so shaped as to be generally s-shaped and thus have rounded edge-portions situated in the region of transition between the central length and the inlet and outlet passages.

7. A cuvette as claimed in any one of claims 1 to 4 in which said edge(s) are formed by a slot in a single centre plate.

8. A cuvette as claimed in any preceding claim being a multiple cuvette comprising more than two plates placed parallel to each other and at least two pairs of opposing faces of the plates having said transparent piece or pieces therebetween.

9. A cuvette as hereinbefore described with reference to the accompanying drawings.

10. A method of producing a flow-through cuvette having a continuous passage comprising a central length and inlet and outlet branch passages which diverge from the longitudinal axis of the central length and extend from the central length to the surface of the cuvette which comprises so shaping a piece or pieces of transparent material and sealingly mounting and sandwiching the piece(s) between plates that opposing shaped edges of the transparent piece(s) define the continuous passage.

11. A method as claimed in claim 10 wherein said edge(s) are shaped so as to provide a gradual transition between the central length and the inlet and outlet passages.

12. A method as claimed in claim 10 or 11 wherein said edge(s) are arranged in a common plane and spaced a predetermined distance apart with their shaped edges facing each other.

13. A method as claimed in claim 10 wherein said edges are formed by cutting out in a single centre plate a slot corresponding to the desired longitudinal profile of the passage and rounding off the edges of the slot so as to provide a gradual transition between the centre length and the inlet and outlet passages.

14. A method as claimed in claim 13

which includes the further steps of sealingly mounting and sandwiching a central plate so formed between plates and cutting the resultant composite block so as to expose the openings of the inlet and outlet passages.

5 15. A method as claimed in any of claims 10 to 14 for producing a multiple cuvette which comprises arranging more than two plates parallel to each other and sealingly
10 mounting and sandwiching a transparent shaped piece or pieces between at least two pairs of opposing faces of the parallel plates.

16. A method as claimed in claim 15 wherein said piece or pieces are sealingly

mounted and sandwiched between each 15
opposing face of more than two plates placed parallel to each other.

17. A cuvette whenever made by a process according to claims 13 and 14.

For the Applicants

D. W. ANGELL

Chartered Patent Agent

Rohm & Haas Company

European Operations

Chesterfield House

Barter Street

London WC1A 2TP

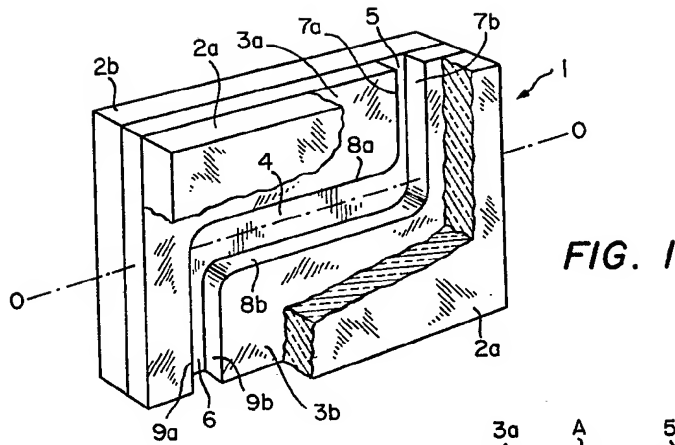


FIG. 1

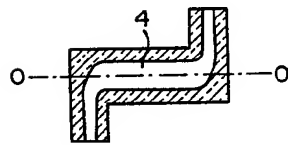


FIG. 3a

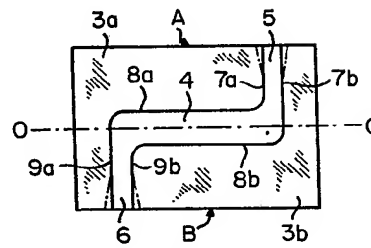


FIG. 2

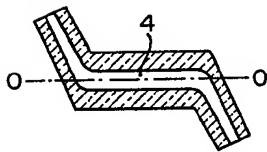


FIG. 3b

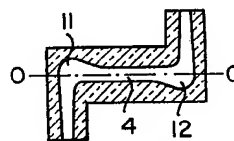


FIG. 3c

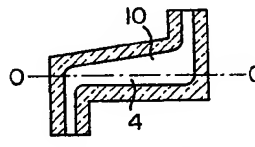
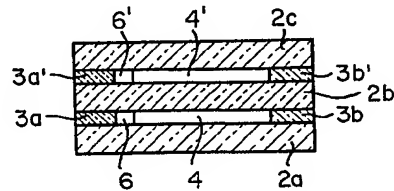


FIG. 3d

FIG. 4



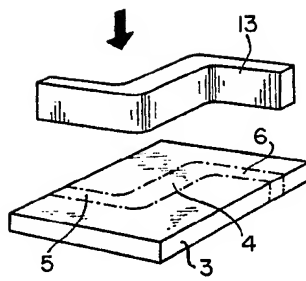


FIG. 5a

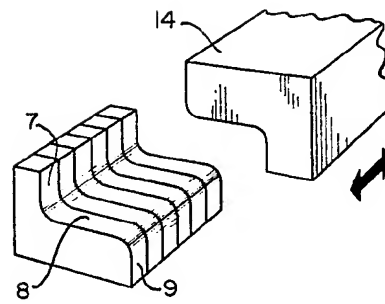


FIG. 5b

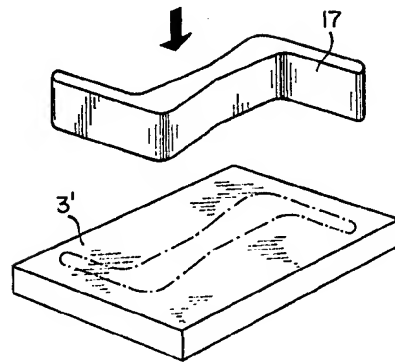


FIG. 6a

FIG. 6b

